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EBSD Analysis of Materials Utilizing High Temperature Protochips Aduro System in FE-SEM

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In recent years with the advances in both EBSD and FE-SEM technology there have been renewed efforts at analyzing nanostructured materials at high temperatures using dedicated specimen holders and sub-stages. Although the techniques for EBSD analysis of bulk materials using heating stages have been well established [1], the requirements for nanostructured materials preparation and analysis obviously differs from bulk materials and can benefit from a miniaturized heater with smaller sample/higher temperature capacity capability [2].

We have performed initial testing of a Protochips Aduro heating and electrical biasing system using a JEOL JSM-7100FT/LV FE-SEM microscope. The holder was redesigned from the original holder configuration to allow smooth transition from flat specimen experiments to 70 deg tilt required for EBSD acquisition, with no geometrical restrictions in terms of standard EBSD working distance selection or EBSD camera insertion. The holder can be inserted seamlessly through the load-lock loading mechanism, while the existing microscope stage has an adapter that connects to electrical feedthroughs that can apply a bias for heating or electrical experiments. The small heating element on the Aduro system enables fast thermal ramp rates and high thermal stability, so the specimen size is limited to approximately 50 μm and smaller, sufficient for powders and other nano-material host specimens.

Our initial set of experiments was performed using an electrical contact, where a thin film of Au (400Å) was deposited on to of existing Ti layer (100Å). The initial Au film structure showed nano-grained material with grain size on the order of 20-100 nm (Fig. 1a). The specimen was tilted to 70deg; a small electrical current was passed through the specimen (14.5 mA) until the sufficient amount of current induced a break in the film and a significant change in Au grain structure (Fig. 1b). Fig. 2 shows the EBSD map of the break area taken at 20kV. This set of data demonstrated the potential of this holder to induce structure transformation which can be characterized via EBSD analysis.

In this paper we will present additional examples of materials analyzed via FE-SEM and EBSD, for example Cu/Ag alloy powder, which has demonstrated dramatic transformation under increased temperature conditions (Fig. 3). Our data shows that the Protochips Aduro heating and electrical biasing system with its fine control of the temperature, the ability to reach temperatures as high as 1200 ºC and electrical biasing capability, has proven to be a viable alternative to traditional bulk holders for the analysis of nanostructured materials using EBSD.

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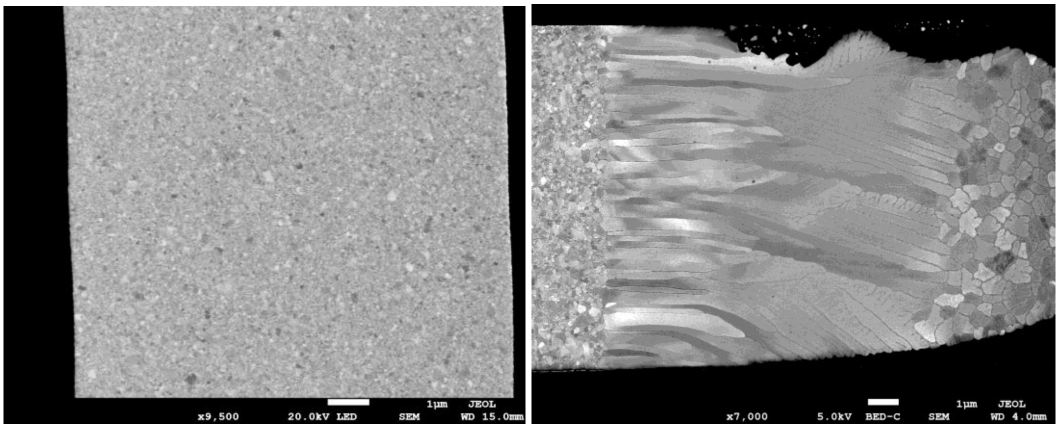

Figure 1. Au (400A) film on Ti – electrical contact. (a, left) before and (b, right) after electrically induced break (image rotated 90 deg from the original orientation).

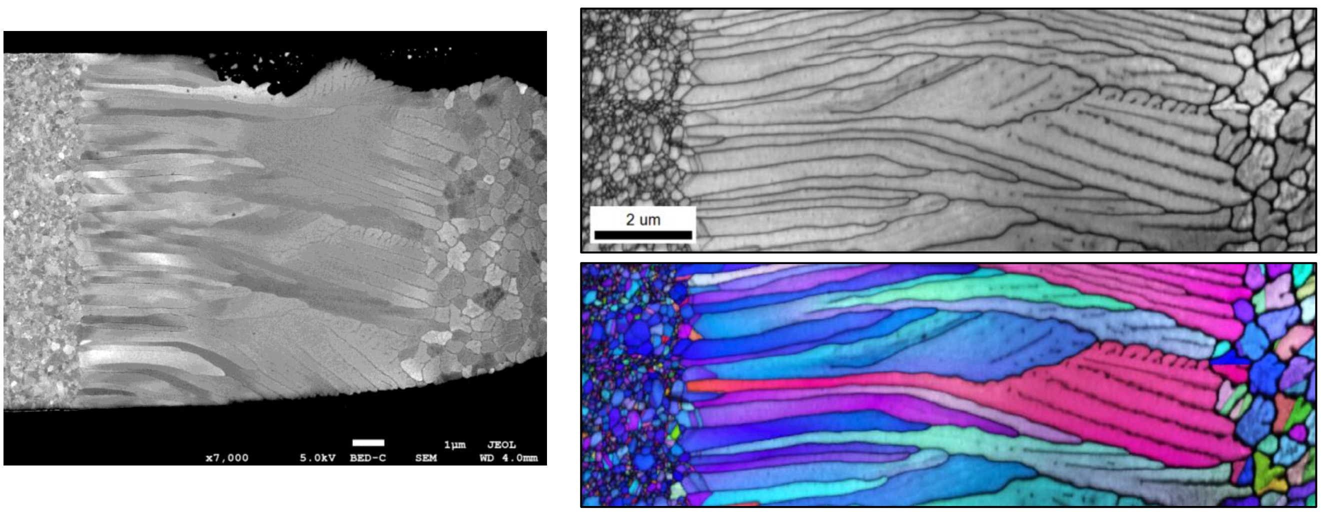

Figure 2. EBSD data collected after electrically induced break. Top - image quality map, bottom – IPF (ND) map. 15kV, 6nA.

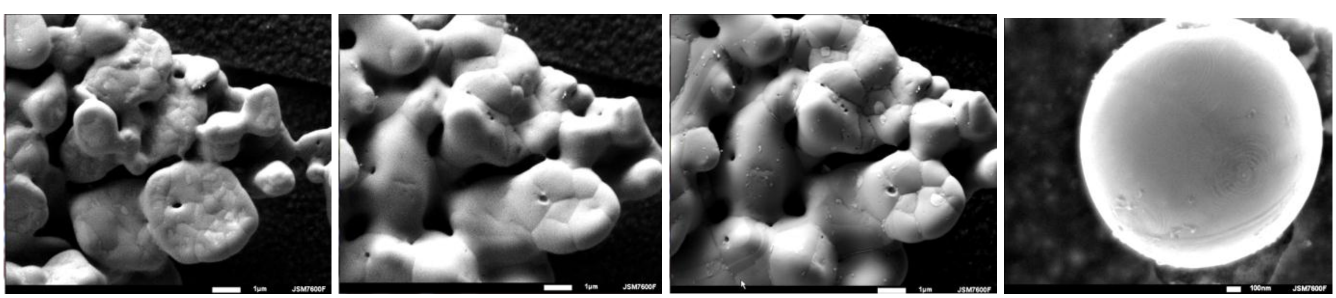

Figure 3. Transformation of Cu/Ag powder under temperature (increasing from left to right 500->650->800->1200C). Note grain coalescence and growth as a result of elevated temperature and eventual result of a 'molten' ball of the alloy. Upon quenching, the ball develops faceting.

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